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IS 12335 (1988): Method for determination of propoxur residues in food commodities [FAD 1: Pesticides and Pesticides Residue Analysis]

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“RE-AFFIRMED 1994” **IS : 12335 - 1988**

(Reaffirmed 2004)

Indian Standard

METHOD FOR DETERMINATION OF PROPOXUR RESIDUES IN FOOD COMMODITIES

0. Foreword — Propoxur formulations are extensively used in agriculture for the control of a broad spectrum of insects. This standard will enable the health authorities and others engaged in the field to follow uniform test procedure for the estimation of propoxur and its major metabolite isopropoxy phenol residues in various food commodities.

1. Scope — This standard prescribes the spectrophotometric method for the determination of propoxur (*O*-isopropoxyphenyl *N*-methyl carbamate) and its major metabolite isopropoxy phenol residues in food commodities.

1.1 The method is applicable with the limit of detection of 0·5 μg/g (0·5 ppm).

1.2 Though no set procedure for thin layer chromatography (TLC) is being prescribed, standardized TLC procedures may be followed, if necessary, for the purpose of clean up, identification and confirmation of propoxur and isopropoxy phenol residues.

2. Quality of Reagents — Unless specified otherwise, pure chemicals and distilled water [*see* IS : 1070-1977 ‘Specification for water for general laboratory use (*second revision*)’] shall be employed in tests.

Note — ‘Pure chemicals’ shall mean chemicals that do not contain impurities which affect the results of analysis.

3. Sampling — The representative samples for the purpose of estimating propoxur residues shall be drawn in accordance with IS : 11380 (Part 1)-1985 ‘Methods of sampling for determination of pesticides residues: Part 1 Agricultural and food commodities’.

4. Preparation of Sample

4.1 *Apparatus*

4.1.1 *Rotary vacuum evaporator*

4.1.2 *Waring blender* — Fire and explosion proof.

4.1.3 *Water bath*

4.1.4 *Chromatographic column* — 400 mm long and 20 mm i.d. with integral reservoir.

4.2 *Reagents*

4.2.1 *Acetone* — All glass redistilled.

4.2.2 *Acetonitrile* — All glass redistilled.

4.2.3 *Hyflo super-cel*

4.2.4 *Methylene chloride* — All glass redistilled.

4.2.5 *Phosphate buffer* (*p*H 5) — Prepare by adding 15 ml of 2/15 M sodium hydrogen phosphate ($NaHPO_45H_2O$) to one litre of 2/15 M potassium dihydrogen phosphate (KH_2PO_4).

4.2.6 *Florisil* — 60-100 mesh, analytical reagent grade. Heat at 130°C to remove moisture. Deactivate by adding 2·5 percent water (2·5 ml water and 97·5 g dried florisil) and let equilibrate for 24 hours in tightly stoppered bottle before use.

4.3 *Extraction* — Transfer 50 g of sample to a blender jar. Add 5 ml water and 200 ml acetone, and blend for 5 minutes at high speed. Filter sample through 12·5 cm Whatman No. 42 or equivalent filter paper covered with approximately 6 mm layer of hyflo super-cel in Buchner funnel. Return filter cake to blender and add 200 ml methylene chloride. Blend the sample at high speed for 3 minutes and filter again. Wash the blender jar and filter cake with 100 ml acetone and 100 ml methylene chloride. Evaporate filtrate to dryness on a rotary vacuum evaporator in 38°C water to bath. Transfer the residue in flask to 250 ml separatory funnel with 50 ml methylene chloride and 25 ml phosphate buffer (*p*H 5). Shake separatory funnel for 30 seconds and let stand for 5 minutes. Drain methylene chloride layer into 250 ml Erlenmeyer flask and extract buffer solution twice more with 50 ml portions of methylene chloride. Combine all methylene chloride phases in flask and evaporate the extract to dryness in rotary vacuum evaporator. Transfer residue in flask to 500 ml separatory funnel using 150 ml hexane and 40 ml acetonitrile successively. Shake separatory funnel for 30 seconds and

Pesticides Residues Analysis Sectional Committee, AFDC [Ref : Doc : AFDC 56 (2576)]

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drain acetonitrile layer into round bottom flask. Add 40 ml fresh acetonitrile to first separatory funnel and repeat second stage extraction. Evaporate combined acetonitrile phases to dryness and preserve residue for clean-up.

4.4 *Clean-Up* — Tap glass wool plug into bottom of chromatographic tube with integral reservoir of methylene chloride. Slowly sprinkle in 15 g florisil and let settle. Tap column with anhydrous sodium sulphate up to 2 cm and drain solvent to top of column. Transfer residue in flask to column with 4 washes of 5 to 10 ml methylene chloride. Elute column with total volume of 250 ml methylene chloride including washes. Transfer methylene chloride elute to rotary vacuum evaporator and evaporate just to dryness. Dissolve residue in appropriate quantity of methanol and estimate propoxur residue as described in **5**.

5. Spectrophotometric Method

5.1 *Principle* — The method is based on the hydrolysis of propoxur with methanolic potassium hydroxide to its phenol and coupling with diazotized 4, 4-diaminodiphenyl sulfone. The orange complex formed has an absorption maximum at 500 nm.

5.2 *Apparatus*

5.2.1 *Spectrophotometer* — with 1 cm cell.

5.3 *Reagents*

5.3.1 *Propoxur* — 99 percent pure.

5.3.2 *Methanolic potassium hydroxide* — 1 N.

5.3.3 *Sodium nitrite* — 1 percent solution in water.

5.3.4 *4, 4-Diaminodiphenyl sulfone* — 0·5 percent solution in methanol.

5.4 *Preparation of Standard Curve*

5.4.1 Pipette 0·5 ml portions of 4, 4-diaminodiphenyl sulfone to clean, dry test tubes. Diazotize by adding 1 ml of 1 percent sodium nitrite solution and 1 ml of 1 N hydrochloric acid to each test tube.

5.4.2 Add 0·05, 0·1, 0·2, 0·4, 0·8 and 1·0 ml aliquots of propoxur working standard solution (5-100 µg) into separate test tubes. Add 2 ml of methanolic potassium hydroxide and 2 ml water to each tube. Shake well and keep tubes on boiling water bath for 30 minutes. Remove tubes and let stand for 10 minutes at room temperature. Quantitatively transfer diazotized reagent (*see* **5.4.1**) into tubes containing hydrolyzed propoxur. An orange complex with an absorption maximum at 500 nm will form immediately (colour stable for 12 hours). Let stand for 5 minutes for full colour development. Quantitatively transfer coloured solution to 20-ml volumetric flask and dilute to volume with methanol. Read absorbance of orange complex at 500 nm in spectrophotometer against blank similarly prepared. Plot concentration of propoxur (0·25-5·0 µg/ml) against absorbance to get a straight line.

5.4.3 *Estimation of propoxur residues* — Dissolve the residue obtained in **4.4** in methanol and transfer quantitatively to a test tube. Proceed as in **5.4.2** for development of orange coloured complex. Quantitatively transfer the coloured solution to a 20-ml volumetric flask and make up the volume with methanol. Read absorbance of this complex at 500 nm against same blank. Read off from the standard curve obtained in **5.4.2**, the concentration of propoxur (C_s) in µg/ml of the sample solution.

5.5 *Calculation*

$$\text{Propoxur content in } \mu g/g = \frac{C_s \times V_1}{1\,000} \times \frac{1\,000}{m} = \frac{C_s \times V_1}{m}$$

where

C_s = concentration in µg/ml of propoxur in sample solution,
V_1 = volume in ml of the sample solution taken for extraction, and
m = mass in g of the sample taken for test.

EXPLANATORY NOTE

In the preparation of this standard, due consideration has been given to the limits of propoxur residues laid under the provisions of *Prevention of Food Adulteration Rules,* 1965 and the test method is sensitive to the prescribed level of residues.

In reporting the result of a test or analysis made in accordance with this standard, if the final value, observed or calculated, is to be rounded off, it shall be done in accordance with IS : 2-1960 'Rules for rounding off numerical values (*revised*)'.

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